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(2 pages total)

# METHOD FOR MANUFACTURING HIGH-PURITY METAL

## Brief Description of the Drawings

The drawings illustrate an example of the apparatus for implementing the method of the present invention. Figure 1 is a horizontal cross section along the B-B line in Figure 2, and Figure 2 is a vertical cross section along the A-A line in Figure 1.

## Detailed Description of the Invention

The present invention relates to a method for manufacturing high-purity metal by using a plasma jet to cause a material to be reduced (such as a silicon halide or titanium halide) to undergo pyrolysis or a reaction with hydrogen.

With conventional methods for manufacturing high-purity metal, the reaction was limited to a relatively low temperature [in order to avoid] contamination from the reaction vessel or the softening and melting of the vessel material. Consequently, the reaction yield and the reaction velocity were both low. Furthermore, since the reaction was usually conducted at a temperature under the melting point of the precipitated metal, the precipitate had to be removed in solid form [from the reactor]. Due to this restriction, the reaction was not generally conducted continuously. In addition, the reactor was inevitably bulky for the amount of material it produced.

Advantages to the method of the present invention include the following:

1. Because the reaction is conducted at an extremely high temperature, the reaction velocity is high, and the raw material yield is good.

2. Because the reaction precipitate is taken off in the liquid phase, continuous operation is possible.

3. Because the portion of the reaction vessel where the product is precipitated is cooled, and the surface thereof is entirely covered with low-temperature precipitate, no contamination from the vessel will be caused by outflow of the liquid precipitate.

4. The apparatus is more compact than in the past.

Next, the apparatus used to implement the present invention will be described in detail through reference to the example shown in the drawings. In Figures 1 and 2, a doubled-walled[1] copper vessel 1 is cooled by cooling water flowing in from [a port] 3 and out of [a port] 4. Electrodes 2 provided to the vessel 1 are made of a metal such as tantalum and are connected to a DC power supply 6 via a terminal 7. When the electrode gap is adjusted and an electrical load applied, an arc is generated between the electrodes. Thoroughly refined hydrogen gas is then introduced from [a port] 5, at which point the arc becomes an extremely high temperature plasma jet that is sprayed into the vessel 9. A material that has been thoroughly refined and is to be reduced is then introduced from [a port] 8 and brought into the jet in the form of a gas or liquid, whereupon these [materials] are mixed in the jet flow and either pyrolyzed or reduced into high temperature hydrogen by the high temperature of the jet, which results in the targeted metal being precipitated. The metal produced here is in the form of particulate gas, but since the walls of the vessel 9 onto which the jet is sprayed are being cooled by water, the jet flow touches and is cooled by these walls, and the precipitated metal forms a solid on the walls and adheres thereto. As this operation is continued, liquid metal ends up being deposited on the walls and flows out from a port 10. The outflowing metal is cast into a mold and solidified. Any unreacted gas and the reaction product gas are discharged through [this port] 10 to the outside of the vessel.

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|---|---|
| Arc voltage: | 150 V |
| Arc current: | 130 amperes |
| Hydrogen flux | 50 L/min |
| Refined silicon tetrachloride flux: | 35 g/min |

A 2.5 kg block of silicon was obtained using 25 kg of silicon tetrachloride under the above conditions. This silicon had a purity of about 99.9999999[%], which was more than good enough for a semiconductor material.

[1] Translator's note: Literally "two-coat" in the original, but the intended meaning seems clear from the context.

## Claims

1. A method for manufacturing high-purity metal, characterized in that a plasma jet is generated by a standard method using refined hydrogen, a metal material that has been refined and is to be reduced is introduced into this plasma jet flow and either pyrolyzed or reacted with hydrogen, causing said metal to precipitate, and this [metal] is liquefied in a cooling vessel and allowed to flow out.

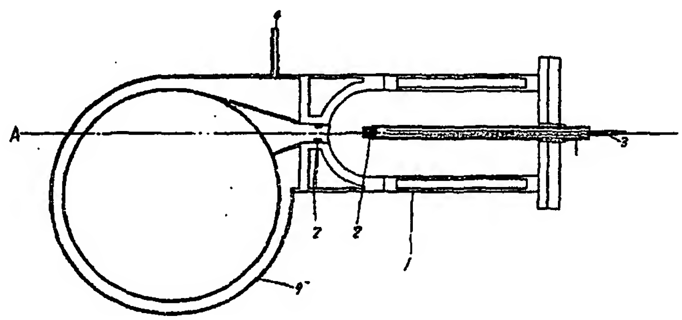

Figure 1

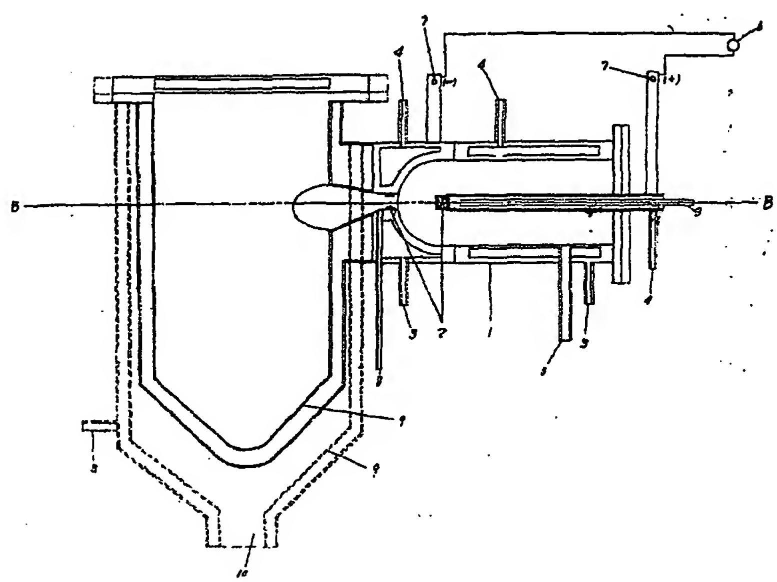

Figure 2

特　許　庁　　　　特許出願公告

# 特許公報　　昭38－6854

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## 高純度金属の製造法

### 図面の簡単な説明

図面は本発明の方法を実施する装置を例示したもので第1図は第2図のB－B線における横断平面図、第2図は第1図のA－A線における縦断側面図。

### 発明の詳細な説明

本発明はプラズマジエツトを使用し被還元材料（けい素ハロゲン化物、チタンハロゲン化物等）を熱分解又は水素と反応させ高純度金属の製造法に関するものである。

従来の高純度金属の製造方法は反応容器からの汚染又は容器材質の軟化融解のため反応温度が比較的低温度に制約されている。従つて反応収率が低く且つ反応速度も小であつた。更に、析出金属の融点以下の温度で反応させることが多いため析出物を塊状で取り出す関係上反応は装置から制約をうけて一般に不連続に行われている。又生産量に比し装置は大型のものとならざるを得なかつた。

しかるに、本発明の方法の利点は

1　反応が極めて高温において行われるため反応速度が速く且つ原料収率が良好である。

2　反応析出物が液相で取り出されるため連続操業が可能である。

3　反応容器の生成物析出部を冷却し、その表面を低温度の析出物で全面的に被覆するから析出物が液状に流出しても容器からの汚染はない。

4　装置は従来のものに比し小型である。

次に、図面に一例として示した本発明を実施する装置について詳細に説明する。第1図及び第2図において、二重壁よりなる鋼製容器1は3より流入4より流出する冷却水によつて冷却されて居る。容器1に付属する電極2はタンタルその他の金属であつて端子7を経て直流電源6に接続されている。電極間隔を調整し、電力を負荷すると電極間にアークが発生する。次いで5より充分に精製された水素ガスを流入させるとアークが極めて高温のプラズマジエツトとなつて容器9内に噴出する。次いで8より充分に精製された被還元材料を気態又は液態でジエツト中に送入すると、これらはジエツト気流中に混入し、ジエツトの高温に依り熱分解又は高温の水素に依り還元され目的の金属を析出生成する。この時の生成金属は微粒蒸気状であるがジエツトの噴出する容器9の壁面が水冷されているからジエツト気流はこの壁面に触れ冷却され析出金属は壁面に固体となつて析出附着する。更に操作を継続すると壁面に液態の金属が析出するに至り流出口10に流出する。流出金属は鋳型に鋳込まれ固化せられる。未反応気体及び反応生成気体は10を経て容器外に排出される。

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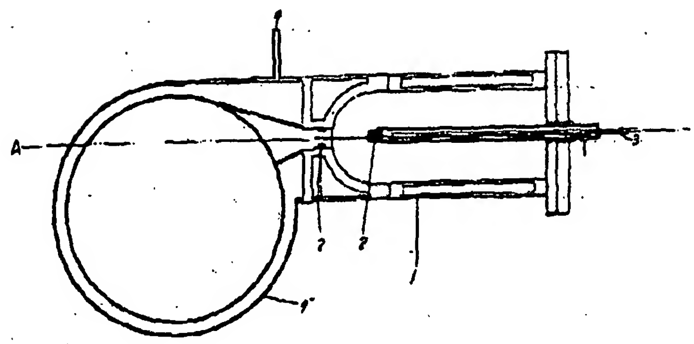
|---|---|
| アーク電圧 | 150V |
| 電流 | 130アンペア |
| 水素流量 | 50ℓ/min |
| 精製四塩化けい素流量 | 35g/min |

以上の条件で四塩化けい素25Kgを使用し2.5Kgの塊状シリコンを得た。このものは99.9999999％程度の純度をもち半導体用材料として充分なものであつた。

### 特許請求の範囲

1　精製された水素を使用し通常の方法によつてプラズマジエツトを発生させ、このプラズマジエツト気流中に精製された被還元金属材料を導入し、これを熱分解または水素と反応させ当該金属を析出させ、これを冷却容器中で液化流出させることを特徴とする高純度金属の製造法。

第1図




第2図